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environmental restoration project

A Department of Energy Environmental Cleanup Program

Environmental Restoration Project
Standard Operating Procedure

for:

X-ray Fluorescence Analysis

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X-ray Fluorescence Analysis

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X-ray Fluorescence Analysis

1.0 PURPOSE

This SOP provides instructions for operation of the Rigaku 3064 X-ray Fluorescence Spectrometer.

2.0 SCOPE

This SOP is a manadatory document and shall be implemented by all ER Project participants when performing X-ray fluorescence analysis with the Rigaku 3064 X-ray Fluorescence Spectrometer for the ER Project.

3.0 TRAINING

3.1 All users of this SOP are trained by reading the procedure. The **users** shall ensure the training is documented in accordance with QP-2.2, and is entered appropriately in the ER Project Training Database located at http://erinternal.lanl.gov/Training/Training.asp.

3.2 The **Geology Task Leader (TL)** will monitor the proper implementation of this procedure and ensure that relevant team members have completed all applicable training assignments in accordance with QP-2.2.

4.0 DEFINITIONS

4.1 *Certified Operator* — Person responsible for analyzing samples and for proper storage and disposition of hard-copy and magnetic media data records. Certified operators are responsible for final disposition and/or storage of samples following analysis.

4.2 *Machine Custodian* — Person responsible for maintenance, including system software and magnetically-stored system back-ups, X-ray counter resolution check, instruction of operators in use of the system, and certification of operators for this procedure. The machine custodian may delegate responsibilities to specific certified operators.

4.3 *NIST* — National Institute of Standards and Technology.

4.4 *PHA* — Pulse height analyzer.

4.5 *XRF* — X-ray fluorescence.

4.6 *XRF System* — X-ray fluorescence system composed of XRF spectrometer, sample changer, and computer hardware and software.

5.0 BACKGROUND AND PRECAUTIONS

This SOP is to be used in conjunction with an approved Group environment, safety, and health plan.

6.0 RESPONSIBLE PERSONNEL

The following personnel are responsible for activities identified in this procedure.

6.1 Geology Task Leader

6.2 Quality Program Project Leader

6.3 Users

6.5 ER Project Participants

7.0 EQUIPMENT

7.1 The XRF system includes a Rigaku 3064 sequential X-ray Fluorescence Analyzer and a Rigaku ASC-24 Sample Changer. These components are controlled by a Digital Equipment Microvax III.

7.2 The software used to operate the XRF system, Rigaku Dataflex Dataflex, is commercially acquired. The VMS operating system is a commercially acquired software package provided by Digital Equipment Corporation. XRF-11 is a commercially acquired software package provided by Criss Software, Inc.

8.0 PROCEDURE

Note: Subcontractors performing work under the ER Project's quality program may follow this standard operating procedure (SOP) for collecting geological samples from cuttings or core, or may use their own procedure(s) as long as the substitute meets the requirements prescribed by the ER Project Quality Management Plan, and is approved by the ER Project's Quality Program Project Leader (QPPL) before the commencement of the activitie(s).

Note: ER Project personnel may produce paper copies of this procedure printed from the controlled-document electronic file located at http://erinternal.lanl.gov/home_links/Library_proc.htm. However, it is their responsibility to ensure that they are trained to and utilizing the current version of this procedure. The author may be contacted if text is unclear.

Note: Deviations from SOPs are made in accordance with QP-4.2, Standard Operating Procedure Development and documented in accordance with QP-5.7, Notebook Documentation for Environmental Restoration Techncial Activities.

The XRF system uses an intense beam of X-rays of specified energy that strike and interact with constituent elements of the target specimen to produce characteristic X-rays of those elements. The characteristic X-rays are detected with a wavelength spectrometer and scaled. The scaled signal is corrected for absorption and fluorescence effects and compared with standard specimens of known elemental concentration. Unknown compositions ranging from trace to major amounts can be quantitatively determined.

Detailed operating instructions for the XRF system are given in the Rigaku Instruction Manuals 1 and 2, the Rigaku Dataflex Software Manual, and the XRF-11 User's Guide, all of which are stored in the XRF laboratory. Specific functions relevant to this procedure are listed below.

8.1 *Intensity Calibration* – A reference disk (always in position 1 of the ASC-24 Sample Changer) is used to correct for instrumental drift. The X-ray intensities from this disk are stored in a file named "Eqstabs.dat." The Rigaku Dataflex Software Manual describes the method for updating these intensities.

8.2 *XRF-11 Standard Reference* – XRF-11 contains standard parameters described both by concentrations and associated intensities. The procedures for utilizing and updating XRF-11 parameters are described in the XRF-11 User's Guide.

8.3 *Detector Resolution Checks* – The Rigaku 3064 contains two X-ray counters, one flow proportional and one scintillation. The resolution checks for the counters are described in the Rigaku Instruction Manuals 1 and 2.

8.4 *Standards* – Certified standards include standards purchased from NIST and standards acquired from other analysts that have been widely distributed and for which composition and homogeneity have been documented in technical publications. An updated list of certified standards can be found in the XRF laboratory.

8.5 *Environmental Conditions* – Normal interior building temperature and humidity are acceptable for the operation of the XRF system. The X-ray tube is cooled by a chilled-water system maintained in the range of 40° to 70°F.

8.6 *Sample Preparation* – Samples will be prepared as fused disks, using a weighed mixture of sample powder and lithium tetraborate flux. Sample powders will typically be prepared by pulverizing samples in a shatterbox or ball mill, although other powders may be used. A split of each powder will be used to obtain loss-on-ignition (LOI) weights whenever sufficient powder is available. Records of sample preparation will be maintained in the XRF sample preparation laboratory.

8.7 *Loss on Ignition and Supplemental Ferrous/Ferric Determinations* – Typical loss-on-ignition (LOI) values reflect loss of water from rock samples; in ironrich samples the LOI determination may be positive as a result of oxidation of ferrous iron. In such samples the LOI data can be supplemented by determination of ferrous iron by titration (Husler, 1989).

8.8 *Sample Control* – Samples will be tracked by fusion log numbers that can be correlated with unique ER project geologic sample identifications.

8.9 *Sources of Error and Uncertainty* – Potential sources of instrumental error or uncertainty beyond those of fixed physical and instrumental limitations will be indicated by the certified operator's inability to generate quantitative analyses of certified standards within accepted two-sigma tolerance limits. Potential errors arising from presence of unexpected or unaccounted for elements will be indicated by inability to achieve totals within a range of 98.5-101.5%.

8.10 *Safety* – Normal operating conditions as performed by certified operators present no safety hazard. Safe operations are covered in the Aug. 31, 2000 Hazard Control Plan for Rigaku 3064 Operation (ID# HCP-EES-1-XRF-1) or in subsequent revisions of this plan as posted on the instrument.

8.11 *Lessons Learned* –

During the performance of work, **ER Project participants** shall identify, document and submit lessons learned in accordance with QP-3.2, Lessons Learned, located at: http://erinternal.lanl.gov/home_links/Library_proc.htm.

9.0 REFERENCES

ER Project personnel may locate the ER Project Quality Management Plan/ER Project QP requirements crosswalk at http://erinternal.lanl.gov/home_links/Library_proc.htm.

The following documents have been cited within this procedure:

QP-2.2, Personnel Orientation and Training

QP-3.2, Lessons Learned

QP-4.2, Standard Operating Procedure Development

QP-4.3, Records Management

QP-5.7, Notebook Documentation for Environmental Restoration Techncial Activities

Criss, J. W., XRF-11 User's Guide, Criss Software Inc., Largo, Maryland, 1985.

HCP-EES-1-XRF-1, Hazard Control Plan for Rigaku 3064 Operation, Aug. 31, 2000.

Husler, J., Determination of Ferrous Iron in Silicate Rocks, UNM Dept. of Geology Procedure, 4 April 1989.

Rigaku Dataflex Software Manual, Danvers, Massachusetts.

Rigaku Instruction Manuals 1 and 2, Danvers, Massachusetts.

10.0 RECORDS

ER Project participants submitting samples for XRF analysis are responsible for submitting the following records (processed in accordance with QP-4.3) to the Records Processing Facility.

10.1 Notebook records of the sample handling and results of analysis relevant to production of XRF data.

10.2 Data submittals for the ER electronic database.

11.0 ATTACHMENTS

None

Using a token card, click here to record "self-study" training to this procedure.

If you do not possess a token card or encounter problems, contact the RRES-ECR training specialist.